5. LOW-COST CARBON FIBER

A. Low-Cost Carbon Fibers from Renewable Resources

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Objective

- Demonstrate the use of new precursor materials that decrease the cost and increase the availability of carbon fiber, which meets the performance and price needs of the automotive market.
- Demonstrate that one or more renewable/recycled precursor formulations can be expected to produce industrial-grade carbon fibers at a cost of $3.00–$4.00/lb.

Approach

- Develop systematically the technical base needed to produce lignin-blend carbon fiber feedstocks at industrial scale:
  1. Produce lignin to obtain best molecular weight, low-volatile, low-salt material.
  2. Use spinning, oiling, and sizing technology.
  3. Use spinning technology, including production die structure, plasticizers, and nucleating agents.
  4. Use plasma treatment and sizing technology to make the fiber compatible with selected resin systems.
  5. Select appropriate polyesters.
  6. Evaluate properties and economics of fiber and composite systems.
- Work with industrial partners to scale and transfer the technology for the production of carbon fiber precursors from lignin blend feedstock.

1. Evaluate melt-extrusion properties of lignin-based feedstock at increasing scale, using near-industrial equipment that can be readily obtained by fiber manufacturers.
2. Evaluate production of carbon fiber using a research production line at an industrial facility.
3. Evaluate mechanical and composite compatibility properties of graphitized melt-spun lignin-blend fibers.
4. Work with partners to better define process economics.

- Transfer technology, including intellectual property, for the production of carbon fibers from lignin to industrial partners.

Accomplishments

- Conducted proof-of-concept demonstration of the melt extrusion of 28-filament tow of lignin-based fibers:
  1. compositions
  2. processing methods
  3. evaluations
  4. fiber quality.
- Indicated by preliminary data that yields of 50%, consistent with those obtained commercially for lignin-based activated carbon, are feasible.
- Successfully spun significant amounts of 28-filament tow at the University of Tennessee (UT) using a two-step process. No sticking problems were apparent, and fiber diameter was reduced from ~45 μm to ~15 μm, with an apparent increase in mechanical properties.
- Conducted preliminary evaluations of a plasma surface treatment plus silanation for lignin-based fibers, which indicated a significant improvement in fiber-resin bonding over conventional carbon fibers.
- Made small epoxy resin composites using carbon fibers produced from UT 28-filament tow, which showed composite mechanical properties.

Future Direction

- Develop methods for production of industrial-quality carbon fibers from lignin blend feedstocks. Studies will include the following:
  1. Optimize lignin preprocessing to minimize contaminants (salts and particulates) and provide the best molecular weight.
  2. Develop conditioning and spinning processes that remove water and volatiles prior to fiber production.
  3. Select and design spinning dies that provide the best internal structure.
  4. Select plasticizers and nucleating agents for lignin-polyester fiber blends.
  5. Select and develop techniques for spooling and oiling lignin blend fiber at each step.
  6. Develop methods for surface treating and sizing the surface of carbon fibers to improve compatibility with proposed resin systems. This is particularly critical for chopped fiber–resin composites.
- Work with project partners to
  1. address raw fiber production issues (lignin, preconsumer recycled polyesters, spinning and winding technologies), and
  2. evaluate carbon fiber production from lignin-based multifilament tow using an industrial research process line.
- Transfer technology, including any intellectual property, to industry.

Introduction

This project focuses on development of carbon fibers from high-volume, low-cost, renewable or recycled fiber sources to reduce precursor and processing costs. Use of these materials also decreases sensitivity of carbon fiber cost to changes in petroleum production and in energy cost.

The early stages of this project focused on proof-of-concept demonstration of the production of single fibers from a variety of high-volume natural, renewable, and recycled materials. These studies showed that carbon fiber, which was primarily composed of Kraft lignin, an inexpensive, high-volume wood pulping byproduct, could be blended with small amounts of a variety of polyolefins and polyesters and melt spun to produce single fibers that could be processed using conventional furnacing sequences to yield carbon fibers.

Single fiber experiments showed that lignin-blend fibers were, in many respects, similar to conventional carbon fiber feedstocks. The fibers, spun at North Carolina State University, required a stabilization (oxidation) cycle, followed by carbonization and graphitization (furnacing in an inert atmosphere). Graphite content, measured with X-ray diffraction, increased with increasing graphitization temperature. The yield from this lignin-blend feedstocks was ~50%, and the fibers were dense, smooth, and round. Fiber properties improved if the fibers were stretched during furnacing.

Based on these studies, it was decided to produce larger amounts of lignin-blend feedstock melt extruded as a multifilament tow. Fiber spinning was moved to UT where a twin-screw Leistritz extruder was available. Using this unit, initial production of multifilament tow was achieved. The major problems encountered were due to contaminants in the desalted commercial lignin used and were resolved by (1) separation of particulate and fibrous contaminants from the lignin, (2) development of a two-step blending and extrusion process, and (3) installation of a filter on the extruder.

This year, larger amounts of multifilament tow were produced using lignin-based feedstocks. In December 2002, 28-filament tow was produced using the Leistritz extruder. As earlier, no interfiber sticking was observed, and small diameter fibers could be produced and spooled. A shear die similar to those used for production of pitch fibers was used to modify the internal structure of the lignin blend fibers. A pre-consumer recycled polyester was used in the lignin fiber blends. Methods for modifying the fiber surface to increase adherence of resin to the fibers were evaluated and used in the successful production of small epoxy resin–carbon fiber composites.

Project Deliverables

By the end of this multiyear program, production of one or more environmentally friendly, economically feasible carbon fiber precursors will be demonstrated, and transfer of production technologies and related intellectual property to industry will be initiated.

In fiscal 2003, two major milestones, the extrusion of 28-filament lignin-blend fiber tow (11/2002) and the production of small resin-fiber composites (9/2003) were completed on time.

Planned Approach

Production of industrial-grade carbon fibers from a radically new type of feedstock requires the simultaneous development of methods for feedstock recovery, preparation, blending, spinning, handling, and spooling in addition to the furnacing, stretching/orientation, oiling, and sizing technologies required for conventional fibers.

Because of high levels of emissions and costs typically associated with spinning of fiber from liquids, first priority was placed on development of melt-spinning techniques for fiber. Use of lignin and other nonnitrogenous feedstocks was preferred because it would eliminate cyanide emissions during furnacing. Use of modern furnacing techniques, such as hot-stretching and controlled atmosphere processing, are being evaluated to improve properties and yield of carbon fiber precursors from feedstock.

After lignin is dried, desalted, and, if necessary, further purified, lignin blend feedstocks will be extruded in a two-step process. First, lignin and a small amount of polyester are blended and extruded as pellets. The pellets are later extruded as small tow (25 to 50 fibers) and will be stabilized and carbonized to permit evaluation of finished fiber structure and mechanical properties. Because the fiber is intended to be used in chopped fiber composites, the fiber will be also be surface treated and sized to increase compatibility of the resin with the fiber surface.

Industrial partners are increasingly involved in the development of process technologies. They have been working with project staff on the selection of blending polymers for lignin, purification of lignin, strategies for production of cleaner lignin, and spinning of fiber. In the later stages of the project, industrial partners will also assist in production of lignin-based carbon fiber, using a research industrial production line.

Transfer of project technology, including any intellectual property, is planned.

Precursor Evaluation

Lignin is an inexpensive, high-volume byproduct of Kraft pulping to produce paper. For lignin to be used as a fiber feedstock, it needs to be purified to remove materials such as particulate, cellulosics, volatile, and pulping chemicals that would interfere with the formation of satisfactory carbon fibers. Byproduct lignin derived from the Kraft pulping process is currently produced as a free-flowing spray-dried powder and is used in a variety of different industrial applications.

Project staff has been working with MeadWestvaco, a project partner which is the major domestic producer of Kraft lignin, to evaluate methods for lignin purification. As commercial lignins contain significant quantities of salt, desalting was the first type of purification investigated. At present, lignin for experimental use is desalted by washing with distilled water and is then dried for experimental use.

During FY 2002 experiments with the UT twin-screw extruder, it was noticed that dies became clogged during extrusion. The 28-filament dies used in extrusion had an average hole size of 225 μm, and future multifilament dies are expected to be smaller. Raw desalted lignin was screened, and contaminants ranging from sand grains and diatoms to nonmelting cellulosic fibers were identified. It was determined that these materials could be decreased significantly by: (1) using a two-dimensional (2-D) sieving technique to remove the bulk of the material from lignin and (2) adding a sandpack filter to the extrusion train.

The hardwood lignin used in current experiments is a finely divided powder. It will naturally contain small amounts of wood-derived volatiles that partition into lignin from pulping liquors. Lignin also sorbs water. Because the extrusion temperature is above the boiling point of water and many volatile organics, single-step extrusion of lignin blends results in the formation of bubbles within the fibers. To decrease bubble formation, a two-step extrusion process has been developed. In this process, lignin and polyester powders are mixed, melted, and extruded to form pellets. Water vapor and volatiles are substantially removed during the pellet extrusion, and the pellets are re-extruded to form fibers. This method was used in spinning the fibers used for composite tests and was effective in preventing the development of most large bubbles. Small angle X-ray examination of fiber bundles does, however, indicate the presence of submicron bubbles in fibers.

If the lignin polyester blend is held in the extruder at high temperatures (>225°C) for extended periods, char formation has been noticed. Examination of lignin powders by nuclear magnetic resonance (NMR) indicates that the charring may be due to short chains of beta-linked sugars attached to the lignin. As the sugars have only small amounts of carboxyl groups, they appear to be derived from a lignin-carbohydrate complex in the wood, rather than from repolymerization of carbohydrates in pulping liquors.

The project staff is currently evaluating the simple industrially feasible methods for carbohydrate removal from lignin.

Working with Eastman Chemical Company staff members, the project staff has been able to use high-volume preconsumer recycled polyesters in feedstock. This is providing a completely renewable and recycle-derived carbon fiber feedstock.

Fiber Spinning

Fiber spinning technology was improved in several different ways during this fiscal year. As described above, the feedstock for fiber was improved by removal of contaminants from lignin and by addition of preconsumer recycled polyesters. The spinning process was modified as a two-step process; pellet extrusion followed by fiber extrusion significantly decreased voids in the fiber.

As described above, a sandpack filter was added to the Leistritz extruder at UT. This is a conventional industrial technology that decreases plugging of the extrusion die.

However, the most significant modification to the fiber spinning process was a change in the spinning die design. In single-fiber extrusion, an untapered cylindrical die was used. This provided dense, smooth-surfaced fibers but did not provide either improved polymer mixing or orientation. In early experiments using the UT Leistritz extruder, the internal fiber structure was dense and without "onion-skin" layers. However, the dies used in fiber extrusion were tapered textile dies that provided minimal mixing and did not alter the structure of polymers within the fiber.

Microscopic examination of surface fracture patterns on ends of fibers broken during mechanical property testing showed that lignin-based carbon fibers did not have an internal structure comparable to that of pitch fibers.

To create a "pitchlike" structure in lignin-blend fibers, a shear die like that used for extrusion of pitch fibers was designed and fabricated. This type of die provides high-shear at the point of fiber extrusion. In addition to ensuring good mixing of fiber components, high-shear mixing increases fiber stiffness. This is shown in Figure 1 and Table 1.

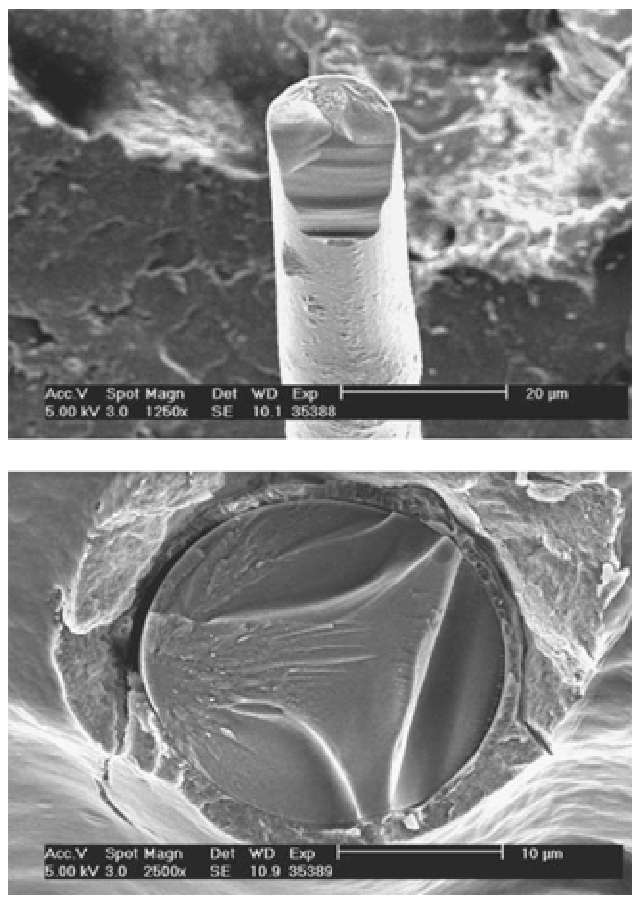

Figure 1. Ends of carbon fiber fracture test specimens showing improved fracture patterns.

Table 1. 1200°C carbonized lignin-blend single-fiber tensile data

Specimen	Diameter (μm)	Peak stress (ksi)	Modulus (Msi)	Strain at peak stress (%)
Spun 11-12/2002 using textile die				
SP2-S4	13.0	125.2	7.9	1.63
Sp2-S5	12.3	110.4	10.3	1.14
Sp2-S7	13.4	149.5	9.2	1.69
Sp2-S9	10.8	141.8	10.1	1.49
Spun 7/2003 using shear die				
Sp5-S2	11.8	149.5	15.8	1.07

Future studies will focus on a combination of die design, spinning conditions, and development of the winding, coating, and spooling technologies required to support technology implementation.

The lignin manufacturer, MeadWestvaco, expects to prepare and store a large batch of hardwood lignin for use by this project.

Fiber Surface Treatment

Lignin fibers are smooth-surfaced and dense. For these fibers to be used in chopped fiber composites, as planned, the surface of the fibers must be modified to increase resin compatibility. In production of conventional pitch and polyacrylonitrile fibers, this is achieved using surface treatments that oxidize the fiber surface and application of a surface coating, or sizing, which improves fiber-resin compatibility. Surface oxidation generally employs wet chemical treatment with strong acids, electrolysis, or a combination of these methods.

Based on earlier experience, it was decided that a combination of plasma treatment of the fiber surface and silanation could provide similar surface treatment with decreased environmental impact. Proof-of-principle tests on small amounts of fiber were completed.

As shown in Figure 2, measurements of fiber surface compositions using an energy dispersive X-ray (EDX) showed significant silanation. This was followed by examination of the retention of fibers in small composite specimens.

The method was scaled up and used for the preparation of small test composites to meet the September milestone.

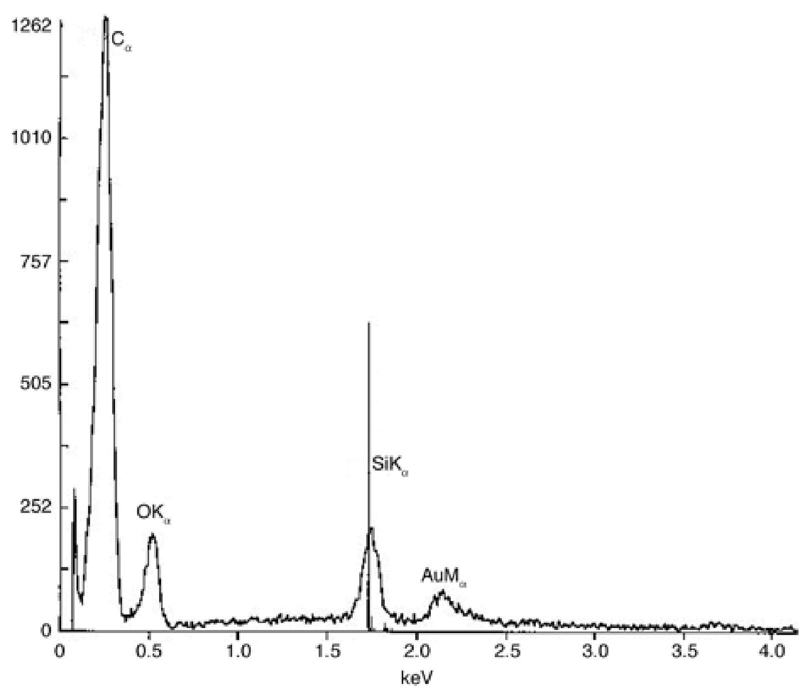

Figure 2. Measurements of fiber surface compositions using EDX showing significant silanation.

As shown in Figure 3, commercial fibers prepared by conventional surface treatment and sizing showed poor adhesion to the resin in small composite fracture tests. However, the plasma treated and silanated lignin-based carbon fibers shown in Figure 4 had excellent retention in the test composite.

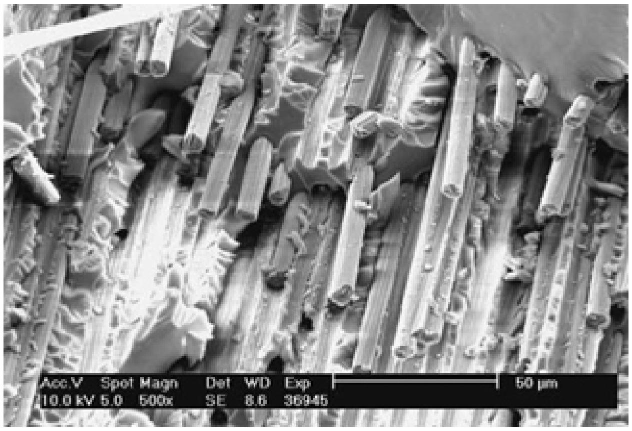

Figure 3. Commercial fibers prepared by conventional surface treatment and sizing showing poor adhesion to epoxy resin and slip out of the resin matrix in small composite fracture tests.

Composite Production

A significant amount of raw lignin-recycled polyester fiber was melt extruded at UT as 28-filament tow and furnaced at ORNL. Some difficulties were encountered due to the fiber being wound as an uncoated textile fiber, rather than as a 30° coated small tow. The need to hand-stretch, fire, and plasma treat individual strands of fiber, rather than a tow, limited the size of samples. The sample is shown in Figure 5. A small composite using an industrial carbon fiber with conventional sizing was also prepared.

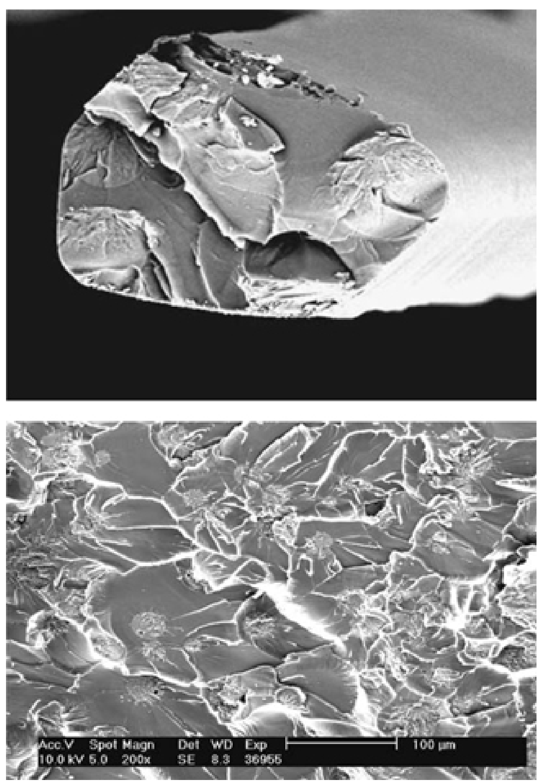

Figure 4. Plasma treated and silanated lignin-based carbon fibers with excellent adhesion to epoxy resin. (*a*) Small test specimen and (*b*) edge of larger composite test specimen.

Figure 5. Small composite prepared from lignin-based carbon fiber.

Evaluation of the composite, a September 2003 milestone, provided an early test to ensure that, further downstream, lignin-blend feedstocks will be useable in resin-fiber composites. To do that, proof-of-concept had to demonstrate these successes: (1) lignin-blend fiber can be melt-extruded as a small tow using near-commercial spinning equipment; (2) composites made from graphitized lignin-blend fibers can be used in resin-fiber composites that have normal fracture patterns, and (3) although very smooth, lignin-blend fibers can be plasma treated and silanated to provide good fiber/resin adhesion.

Future Directions

Significant production of lignin-based multifilament (28-strand) tow and successful use of this material in small resin-fiber composites were demonstrated this year. The project was reproposed and extended to permit evaluation of methods for producing high-quality lignin-based feedstocks for low-cost production of automotive carbon-fiber resin composites.

Having established proof-of-concept, the project staff will systematically address the technical issues required to produce industrial-grade lignin-based carbon fiber at prices and properties meeting automotive need. As the bulk feedstock used in this process is derived from Kraft pulping, a major concern is developing methods for consistently recovering clean, high-quality lignin low in volatiles, water, particulates, and salts. Additionally, the molecular weight of lignin varies considerably with pulping conditions, so delineation of the pulping conditions required to consistently produce industrial-grade carbon fiber will be evaluated.

Spinning die designs for production of lignin-based fibers will be evaluated. Initial tests indicated that dies, similar to those used for spinning pitch-based feedstocks, which provide high shear, create a more uniform internal structure in the raw fiber. Spinning parameters, including rheology, of the blend will be optimized for multifilament production.

Production techniques that provide high-quality, handleable, spoolable raw lignin-based fiber will be required. This will include selection and evaluation of plasticizers and nucleating agents, as well as raw fiber coatings and oils. Spooling techniques will also be evaluated.

After lignin-based fiber is carbonized, it must be surface treated and sized to increase compatibility with the resin system. Fiber-resin compatibility is particularly critical in automotive applications because the current program plan calls for use of chopped, rather than woven or wound, fiber.

Throughout the period, a variety of analytical techniques, including physical property evaluations of fiber and resin, as well as x-ray diffraction and electron microscopic examination, will be used to assess fiber quality.

As studies proceed, project partners expect to become increasingly involved in research activities. These will include tests of the fiber on a research furnacing line of an industrial partner. In 2006, formal transfer of technology for production of carbon fiber from Kraft lignin blend feedstocks, and associated intellectual property, will be initiated.

Partnerships

A number of different entities have been instrumental in development of this technology. North Carolina State University earlier spun a variety of lignin-blend polymers as single fibers. These were used in earlier feasibility evaluations. At present, UT is working to prepare significant quantities of multifilament tow for project use.

The project also benefits from the participation of Eastman Chemical Company, which provides both technical assistance in spinning protocols and significant quantities of polyesters for spinning. Eastman Chemical Company of Kingsport, Tennessee, has also provided preconsumer recycled polymers. Mead-Westvaco at Charleston, South Carolina, has provided hundreds of pounds of lignins, including a wide variety of research lignins, and it has worked closely with the ORNL staff in lignin purification.

Conclusions

During this period, the project staff met all major goals and milestones. Spinning of significant quantities of 28-filament tow from a feedstock composed of Kraft Lignin and preconsumer recycled polyester, was successfully demonstrated. This material was successfully stabilized and carbonized, plasma treated and silanated, and used to prepare small epoxy resin composites.

As a part of this effort, a number of improvements were made to the feedstock production process. These included: (1) characterization of impurities and Kraft lignin feedstocks and development of methods for their removal, (2) development and implementation of a two-step extrusion process that greatly decreased flaws in raw lignin-blend fiber, (3) demonstration of use of preconsumer recycle polyester as the alloying polymer in the raw feedstock, (4) proof-of-principle demonstration of the use of high-shear dies in improvement of lignin-based carbon fiber properties, (5) proof-of-principle demonstration of a plasma surface treatment process that greatly improved adhesion of lignin-based carbon fiber to epoxy resin, (6) preparation of significant quantities of multifilament lignin-based fiber for use in manufacture of small test composites, and (7) preparation and evaluation of the small composites.

The project was retasked, partnerships with Eastman Chemical Company and Mead-Westvaco formalized, and the staff is developing the technical base required to support larger scale production of carbon fibers from high-lignin feedstocks.

B. Low-Cost Carbon Fiber Development Program

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Contractor: Hexcel Corporation
Contract No.: 450001675

Objective

- Define technologies needed to produce a low-cost carbon fiber (LCCF) for automotive applications at a cost of \$3.00 to \$5.00/lb in quantities greater than 1M lb/year. The required carbon fiber properties are tensile strength greater than 400 ksi, modulus greater than 25 Msi, and strain to failure greater than 1%.

Approach

- Develop new precursors that can be converted into carbon fiber at costs below the costs of current processes.
- Explore processing by methods other than thermal pyrolysis.
- Develop technologies leading to significant improvements in current production methods and equipment.
- Develop alternative methods for producing carbon fiber from pitch, polyacrylnitrile (PAN), or other precursors.
- Reduce precursor cost by the use of commercially available energy-efficient precursors and high conversion yields.
- Improve precursor production economics of scale and throughput.
- Introduce novel LCCF production methods.

Accomplishments

- Evaluated proposed research areas through laboratory trials and refinement of manufacturing cost analyses:
  - PAN-based precursors: large-tow benchmark, commodity textile acrylic tow, chemical modifications, acrylic fibers spun without solvents, and radiation and nitrogen pretreatment of PAN-based materials.
  - Precursors other than PAN: polyolefins—polypropylene (PP), linear low-density polyethylene (LLDPE) and high-density polyethylene (HDPE); polystyrene; and polyvinyl chloride (PVC) pitch.
- Scaled-up promising technologies to pilot line trials.
- Assessed the technical and economic feasibility of the proposed research areas.
- Down-selected the most promising technologies to meet the program objectives:
  - Commodity textile acrylic tow with chemical modification or radiation pretreatment.
- Developed detailed manufacturing cost models for the downselected technologies.
- Completed the engineering feasibility studies for large-scale production line.
- Completed the economical analysis to predict product production costs.
- Awarded a short-term (1-year) program to develop carbon fiber roving for the P4 process to meet the immediate needs of the ACC development programs.
- Proposed a Phase II long-term (3-year) program to build on the results of the LCCF program to produce production quantities of commodity textile-acrylic-based carbon fiber for the ACC programs (under review by ORNL).

Future Direction

- Complete the program final report.
- Complete the short-term program for the manufacturing and delivering carbon fiber roving to meet the immediate needs of the ACC development programs.
- Start working on the proposed long-term program (if the program is awarded).
  - Long-term (3-year) program to build on the results of the LCCF program to produce production quantities of commodity textile-acrylic-based carbon fiber for the ACC programs.

Introduction

The goal of this program is to define and demonstrate technologies needed for the commercialization of LCCFs to be used in automotive applications. Lighter-weight automotive composites made with carbon fibers can improve the fuel efficiency of vehicles and reduce pollution. For carbon fibers to compete more effectively with other materials in future vehicles, their cost must be reduced. Specifically, this program targets the production of carbon fibers with adequate mechanical properties, in sufficiently large quantities, at a sustainable and competitive cost of \$3 to \$5/lb.

Project Deliverables

At the end of this multiyear program, technologies for LCCF production will be defined. This definition will include the required materials and facilities and will be supported by detailed manufacturing cost analyses and processing cost models. Laboratory trials and pilot-scale demonstrations will be performed to support the defined technologies.

Planned Approach

This program was divided into two phases:

Phase I: Critical review of existing and emerging technologies, divided into two tasks:

Task I.1. Literature review and market analysis.

Task I.2. Laboratory-scale trials and preliminary LCCF manufacturing cost assessments of the proposed technologies. Phase I led to further refinement and down-selection of the most promising technologies for Phase II.

Phase II: Evaluation of selected technologies using pilot-scale equipment and cost models. Phase II was divided into three tasks:

Task II.1. Pilot-scale design for the evaluation of selected LCCF technologies. This included modifications of a PAN spinning pilot line and two different carbon fiber conversion lines (a single-tow research line and a multitow pilot line) and the construction of continuous sulfonation processing equipment

Task II.2. Experimental evaluation of down-selected LCCF technologies, including commodity textile-tow PAN (with chemical modification and radiation and/or nitrogen pretreatment) and polyolefins (LLDPE and PP).

Task II.3. Large-scale feasibility study of selected LCCF technologies.

Conclusions of FY 2001 Results

(October 1, 2000 to September 30, 2001)

The results of the laboratory trials of proposed technologies were as follows.

1. Further work on acrylic fibers spun without solvents, plasticized PAN, PVC, and polystyrene were halted because of technical, environmental, and cost issues.
2. The following most promising LCCF technologies for Phase II were evaluated and selected: commodity textile PAN-based precursors (as-received and with pretreatment using chemical modification, radiation, and nitrogen prestabilization technologies) and polyolefin precursors (LLDPE and PP).
3. A large-tow PAN precursor technology benchmark was used as a metric to evaluate the proposed technologies in terms of their potential to meet the LCCF program's cost targets. The difference between commodity textile PAN and large-tow precursor, based on carbon fiber cost, is approximately \$1.80 vs \$3.10/lb.

Conclusions of FY 2002 Activities

(October 1, 2001 to September 30, 2002)

These are the highlights of the progress made during FY 2002:

1. Demonstrated the technologies of using chemical modification and radiation pretreatments of commercial commodity textile (28K) tow to produce LCCF that meets the program targeted properties and estimated cost predictions.
2. Developed manufacturing recipes for the conversion of commodity textile acrylic fibers into LCCF using the technologies of chemical modifications and radiation treatments.
3. Developed estimated carbon fiber cost using chemical modification and radiation pretreatments of commercial commodity textile acrylic fibers.
4. Demonstrated the conversion of LLDPE to LCCF that meet the targeted properties of the program and estimated cost

predictions. Due to the issues of sulfuric acids recycling and the available precursor, we concluded that the LLDPE-based technology would need more development efforts to compete with modified textile PAN-based technologies.
5. Commenced the engineering feasibility study of the production lines to produce LCCF based on commodity textile acrylic PAN using chemical modification and radiation pretreatment technologies.
6. Started the plans and statements of work for the long-term and short-term follow-up programs.
7. Updated and refined cost models to reflect the accomplishments in FY 2002.
8. Produced four papers (three published during SAMPE 2002, in Baltimore, Maryland, and one for the Global Outlook for Carbon Fiber 2002, in Raleigh, North Carolina, and a presentation during the SAMPE 2002, in Long Beach, California).

Summary and the Results of FY 2003

Task II.3. Large-scale feasibility study of selected LCCF technologies

During FY 2003 activity was concentrated on task II.3 the large-scale feasibility study for the selected technologies and the economical analysis for predicting product costs. The baseline for this task was a carbon fiber conversion process based on subscale work done during this program using textile acrylic fiber, chemically modified in acrylic fiber manufacturing process, and radiation pretreatment in-line carbon fiber manufacture. The production facility was based on two manufacturing lines in a common building of 2.0 MM lb/year per line. The feed material is 505K, 1.5 dpf textile acrylic fiber. The engineering feasibility study included the following:

1. manufacturing equipment needed for product “recipe,”
2. facility layout and design,
3. material flow and handling,
4. equipment and facility capital costs,
5. manufacturing utility costs,
6. supply of raw materials, and
7. economical analysis of predicted product costs for the selected technologies.

The following is a summary of the design guidelines and processing parameters that we used in this study:

1. production facility based on nominal 2.0M lb/year per line,
2. two manufacturing lines in a common building (baseline case),
3. feed material is 8 tows of 505K at 1.5 dpf, in boxes containing 1500 lb of PAN,
4. final line speed is 420 m/h =1378 ft/h (baseline case),
5. three oxidation ovens (11 passes of 43 ft each, total heated length = 1419 ft, and residence time = 64 min),
6. carbonization (LTF = 30 ft–1.2 min, and HTF = 36 ft–1.5 min),
7. surface treatment using electrochemical (anodic) oxidation,
8. sizing bath (double-roll dip),
9. sizing drying (heated drums), and
10. product take-up (piddle-pack in boxes).

Figure 1 shows the building and equipment layout. Each creel has 2 times 8 positions for continuous feeding, and the facility layout allows a “mirrored” second building to add another 4.0M lb/year.Table 1 gives the estimated facility costs, and Table 2 gives the estimated utility costs. Table 3 gives the estimated product cost for baseline for 2 lines (4.0M lb/year).

Economic Analysis and Cost Model—Baseline Assumptions

1. Raw material

- Textile acrylic fiber $0.80/lb
- Same, chemically modified $0.91/lb
- Box weight 1,500 lb
- PAN length per box 8,089 m (26,540 ft)

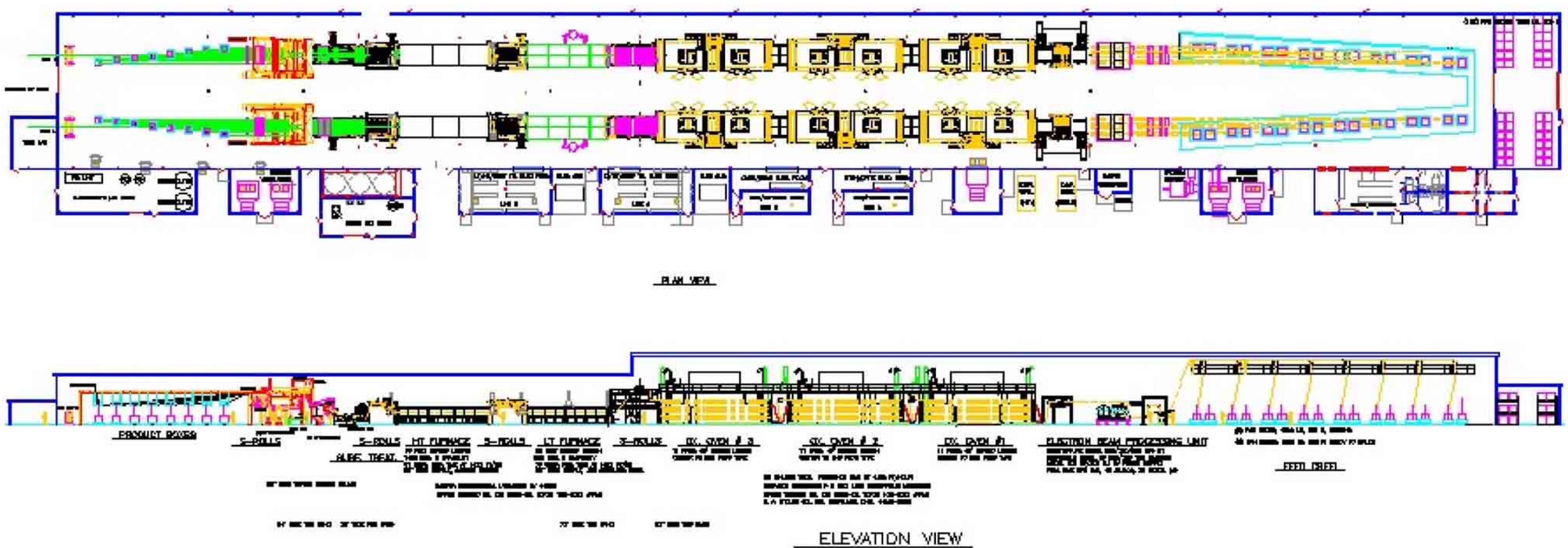

Figure 1. Building and equipment layout.

Table 1. Estimated facility costs

Facility configuration nominal capacity	1 Line (2.0M lb/year)	2 Lines (4.0M lb/year)	4 Lines (8.0M lb/year)
Building	$2,938K	$2,938K	$5,876K
Feed creel and oxidation	$5,601K	$11,055K	$22,111K
Carbonization	$6,162K	$12,199K	$24,397K
Aftertreatment	$2,449K	$3,982K	$7,963K
Engineering costs	$1,630K	$2,119K	$2,771K
Total facility costs	$18,780K	$32,293K	$63,119K
Capital cost (per lb/year CF)	$9.39	$8.07	$7.89
Optional equipment			
Radiation pretreatment	$2,379K	$4,671K	$9,343K
Distributed control system	$1,010K	$2,021K	$4,041K

Table 2. Estimated utility costs

Facility configuration nominal capacity	1 Line (2.0M lb/year)	2 Lines (4.0M lb/year)	4 Lines (8.0M lb/year)
Electricity	$429K	$805K	$1,610K
Natural gas	$446K	$867K	$1,734K
Water	$2K	$3K	$6K
Nitrogen	$106K	$213K	$425K
Steam	$19K	$39K	$77K
Compressed air	$1K	$1K	$2K
Sewer	$1K	$2K	$4K
Total utility costs	$1,004K	$1,930K	$3,859K
Utility cost (per lb/year CF)	$0.50	$0.48	$0.48
Optional equipment			
Radiation pretreatment	$63K	$126K	$252K

Table 3. Estimated product costs (baseline: 2 lines = 4.0M lb/year

Hexcel proposed processes	Large tow precursor		Textile acrylic tow with			
			Chemical modification		Radiation treatment	
Process steps	Mill cost	With 20% ROI	Mill cost	With 20% ROI	Mill cost	With 20% ROI
Precursor ($/lb)	1.59		0.91		0.80	
Precursor ($/lb of CF)	3.53	3.53	2.02	2.02	1.78	1.78
Pretreatment					0.15	0.49
Stablization/oxidation	0.79	1.47	0.79	1.47	0.79	1.47
Carbonization	0.67	1.35	0.67	1.35	0.67	1.35
Sizing, S.T., packaging, Q.C.	0.56	0.81	0.56	0.81	0.56	0.81
SG&A/IR&D	0.40		0.40		0.40	
Total per pound of CF	$5.95	$7.56	$4.43	$6.05	$4.34	$6.30
Changes from baseline	None					

2. **Processing**
   - Product speed 420 m/h (1378 ft/h)
   - Maintenance downtime 15 d/year
   - Production cycle 18 creel loads (~15 d)
   - Downtime per cycle 1.87 d
   - Uptime per year 85.4%
   - Conversion yield 50%
   - Production yield 90%
3. **Labor costs**
   - Average compensation $16.50/h
   - Taxes and benefits 40% of compensation
4. **Staffing** (paid 365/24)
   - Operators (direct labor) 8 (1 line = 6; 4 lines = 14)
   - Technicians (direct labor) 2 (1 line = 2; 4 lines = 3)
   - Indirect labor 40% of direct labor
5. **Labor allocation**
   - Creel—oxidation 35%
   - Carbonization 15%
   - After treatment 50% (includes surface treatment, sizing, packaging, QC)
6. **Other expenses (per year) of invested capital**
   - Capital charge (depreciation) 5%
   - Taxes and insurance 2%
   - Maintenance 3%
   - Return on investment (ROI) 20%
7. **SG&A/IR&D (per year)**
   - 4.0M lb/year on 2 lines (baseline) $1.6M = 10% of mill cost
   - 2.0M lb/year on 1 line $1.2M = 75% of baseline
   - 8.0M lb/year on 4 lines $2.4M = 150% of baseline
8. **Utilities & materials**
   - Utilities rates (based on SLC, UT)
   - Materials (electrolyte, sizing) $0.054/lb of CF
   - Packaging Use PAN boxes

The conclusions of the baseline product costs/price follow:

1. LCCF technologies can reduce product price compared to large tow precursor:
   - Chemical modification by $1.50/lb of CF
   - Radiation treatment by $1.25/lb of CF
2. To achieve a sustainable price of <$5.00/lb, additional measures are needed by implementing the following. Improvements in cost/price are feasible by
   - reduction of invested capital cost,
   - higher line speed,
   - shorter downtime per cycle,
   - longer production cycles,
   - increased throughput over time (learning curve), and
   - higher conversion yield (optimized chemical modification).

Figure 2 shows the impact of implementing these improvement measures on the baseline estimated product cost.

Summary of Task II.3. Large-scale) feasibility study of selected LCCF technologies

1. Defined manufacturing facility and processing conditions.
2. Sustainable price of just below $5/lb of LCCF appears achievable.
3. Cost drivers are
   - precursor (commodity acrylic fiber) $1.94
   - invested capital (ROI) $1.06
   - labor and SG&A/IR&D $0.81
   - utilities and materials $0.48
   - depreciation $0.26
   - maintenance $0.14
   - taxes and insurance $0.10
4. Further reductions in product cost should focus on
   - precursor and capital cost
   - fine tuning other cost contributors

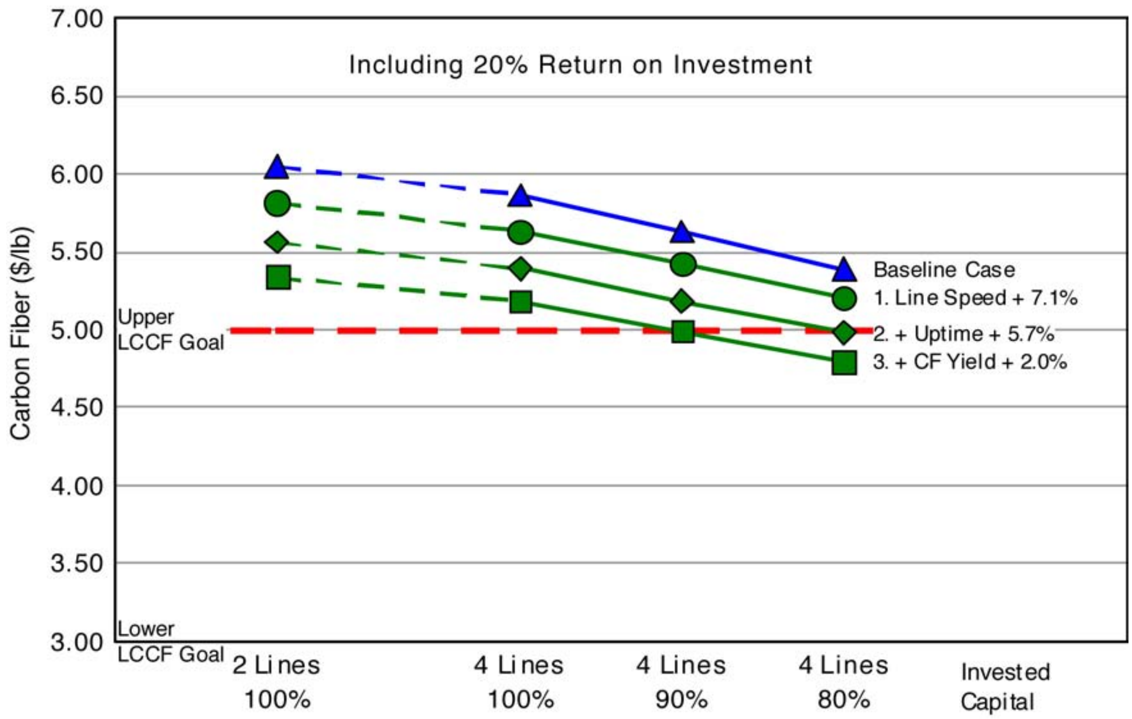

Figure 2. Estimated product cost—improved conversion yield.

Future Work

A Phase II program that is built on the results of Hexcel's LCCF Phase I program was proposed with the following objectives: (1) scale-up and verify the defined technologies and (2) integrate in ongoing automotive research activities by DOE/ORNL and USCAR/ACC. The goals of this future work follow.

1. Develop production technologies utilizing commodity textile acrylic precursor with chemical modification.
2. Identify product forms for automotive applications.
3. Develop and apply suitable technologies to make the identified product forms.
4. Provide needed quantities to support DOE/ORNL programs.

A program plan that addresses the above goals into three major tasks was developed. The following is a summary of these tasks.

Task 1. (Develop plans for the integration of defined technologies for use in on-going research activities by DOE/ORNL and USCAR/ACC

The major milestones for this task follow:

1. Develop integration plan for review and approval by all parties involved.
2. Define the requirements for precursors and carbon fiber to meet the needs of the on-going research activities, that includes the following:
   - precursors for oxidation studies and oxidized/stabilized precursors for microwave processing, and
   - carbon fiber product forms for the automotive industry that include surface treatment, sizing systems, make-up and packaging.
3. Finalize the integration plans and material requirements.
4. Plan a design review meeting to approve the integration plan.
5. Commence implementation of production technologies.

Task 2. (Implement technologies for chemical modification of textile acrylic and its conversion to carbon fiber

The major milestones for this task follow:

1. Implement chemical modification equipment into textile acrylic production line.
2. Modify carbon fiber (pilot) line to handle textile acrylic tow (unsplittable and splittable tows).
3. Develop production procedures for precursor and carbon fiber line operations.
4. Conduct trials to verify Phase I results for precursor and carbon fiber.

Task 3. (Develop and implement techniques to manufacture Product Forms (as defined in Task 1)

The major milestones for this task follow:

1. Support of ongoing research at DOE/ORNL for integration into oxidation studies and oxidized/stabilized precursors for microwave processing.
2. Carbon fiber of various forms to support ongoing research and development by DOE/ORNL and USCAR/ACC, such as continuous tow for prepreg, woven fabrics, and chopped fiber (HexMC).
3. Carbon fiber roving for the P4 process using splittable precursor or carbon fiber tow dividing.

The proposal for this Phase II program for consideration and evaluation was completed and submitted to ORNL. The proposed Phase II program is for 3 years. Hexcel planned this Phase II scope based on input and results of the joint working relationship with Sterling Fiber of Pace, Florida, during Phase I of the LCCF program.

Summary and Conclusions of FY 2003 Activities

These are the highlights of the progress made during FY 2003.

1. We completed Task II.3, the large-scale feasibility study, for the selected technologies and the economical analysis for predicting product costs, based on subscale work done during this program using textile acrylic fiber, chemically modified in an acrylic fiber manufacturing process, and radiation pretreatment in-line carbon fiber manufacture.
2. We proposed a Phase II program that is built on Phase I results with the following goals
   - Scale-up and verify the defined technologies.
   - Define product forms.
   - Develop and implement techniques to manufacture product forms.
   - Integrate results in ongoing automotive research with other activities by DOE/ORNL and USCAR/ACC.

C. Microwave-Assisted Manufacturing of Carbon Fibers

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Contract No.: DE-AC05-00OR22725

Objective

- Investigate and develop a microwave-assisted technical alternative to carbonize and graphitize polyacrylonitrile (PAN) based precursor.
- Prove that carbon fiber with properties suitable for use by the automotive industry can be produced inexpensively using microwave-assisted plasma (MAP) processing.
- Demonstrate that MAP processing can produce acceptably uniform properties over the length of the fiber tow.
- Show that for specified microwave input parameters, fibers with specific properties may be controllably and predictably manufactured using microwave furnaces.
- Most important, demonstrate the economic feasibility for producing approximately 30-Msi modulus fibers at a significant cost reduction relative to those produced conventionally.

Approach

- Demonstrate the ability to deliver high fiber throughput by increasing line speed and tow count.
- Conduct parametric studies on the continuous carbon-fiber processing pilot unit to continually improve the system design, process parameters, and fiber properties.
- Characterize MAP processed carbon fibers to confirm that they satisfy program requirements.

- Continually evaluate, develop, and characterize "spin-off" technology, hardware, and ideas that improve upstream or downstream processing, or facilitate more efficient utilization of fiber.

Accomplishments

- Carbonized 50-K PAN fiber tows were successfully processed at single-tow line speeds up to 200 in./min. This is approximately twice the typical line speed employed in conventional processing. Based on the knowledge and experience gained in this project, the research team believes that processing speeds considerably exceeding 200 in./min are achievable after appropriate system modifications.
- Upgraded the pilot line to simultaneously process three 50-K tows.
- Carbonized 50-K PAN fiber tows successfully at three-tow line speeds up to 12 in./min.
- Discovered a stable three-tow operating mode that requires very little feed gas and yields excellent mechanical properties at 12-in./min line speed.
- Evaluated physical, mechanical, and morphological properties for fiber samples manufactured at single-tow line speeds up to 200 in./min and three-tow line speed of 12 in./min.
- Completed initial software development for an improved dielectric measurement system that can operate from 250°C to >1,000°C with kilohertz sampling rates.
- Issued a patent, entitled "Microwave and Plasma-Assisted Modification of Composite Fiber Surface Topography."
- Performed physical and morphological evaluations to support Argonne National Laboratory's (ANL's) carbon-fiber recycling program, and delivered results to ANL researchers.

Future Direction

- Increase 3-tow line speed to 40 in./min.
- Continue parametric studies and fiber characterization to better understand process effects and processing window, and to quantify fiber properties.
- Achieve integration with other carbon-fiber technology developments, for example, new precursors, rapid oxidation/stabilization processes, advanced surface treatment, advanced downstream formatting, and/or component manufacturing processes (future project).
- Develop and demonstrate related technologies in the area of carbon-fiber manufacturing (e.g., advanced characterization, surface treatment, sensing and control technology, recovery and reuse) as resources and time permit.
- Develop partnership(s) to commercialize the technology.

Introduction

The purpose of this project is to investigate and develop a microwave-assisted technical alternative to carbonize and partially graphitize the PAN precursor. The project is to prove that carbon fiber with properties suitable for use by the automotive industry can be produced inexpensively using MAP processing. It is to be demonstrated that MAP processing can produce acceptably uniform properties over the length of the fiber tow. The project is also to show that for specified microwave input parameters, fibers with specific properties may be controllably and predictably manufactured using microwave furnaces. Finally, but most important, this project is to demonstrate the economic feasibility for producing approximately 30-Msi modulus fibers at a significant cost reduction below those produced conventionally.

Project Deliverables

At the end of this multiyear program, a continuous, multiple-tow, scalable, high-production line speed MAP carbon fiber prototype unit will have been developed, constructed, and tested. A final report will be issued with the test results of the carbon fibers processed with this unit. Appropriate industry briefings will be conducted to facilitate commercialization of this economically enabling technology.

Fiber Properties

A key goal of this project is to demonstrate that carbon fibers with satisfactory material properties can be produced by the MAP process. Program goals established for fiber properties are 25-Mpsi tensile modulus, 250-ksi ultimate tensile strength, and 1.0% ultimate strain. During this reporting period, the project team evaluated the physical, mechanical, and morphological properties of carbon samples that were carbonized and partially graphitized at single-tow line speeds of 130 to 200 in./min, and at three-tow line speed of 12 in./min. Results are compared to commercial fibers that were tested by the same method, as well as to program requirements. Mechanical properties are shown in Figure 1, where 97.7% basis means that 97.7% of property data should exceed the indicated value, and MAP3@12 indicates three tows MAP processed at 12 in./min. Mechanical property results generally satisfied the specified requirements, except that the ultimate tensile strength was slightly deficient at the highest single-tow line speeds. This is attributed to the fiber transport and tensioning system that was not designed for high line speeds. To resolve this problem, a commercial, high-speed, fiber transport and tensioning system was installed during the upgrade to three-tow

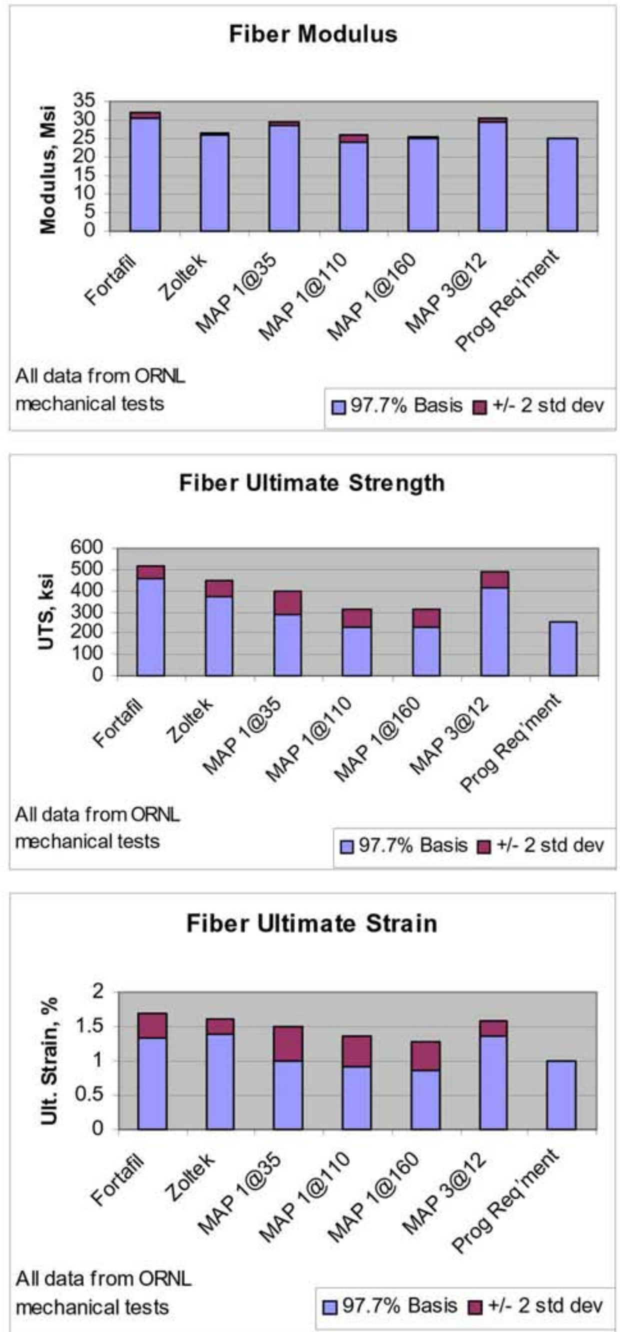

Figure 1. Measured carbon-fiber properties.

capacity. Physical and morphological fiber test results are tabulated in Table 1. Physical properties generally compared favorably with those of commercially available, large-tow commercial-grade fibers.

Fiber morphology was measured by X-ray diffraction. Figure 2 shows plots of intensity vs 2θ for four of the MAP fibers prepared at

Table 1. Physical test data

Fiber type	Production line speed (in./min)	Tow linear electrical resistance (Ω/m)	Electrical resistivity 10^{-3} (Ω-cm)	Calculated filament diameter (µm)	Calculated tow area 10^{-2} (cm^2)	Density (pycnometer) (g/cm^3)
MAP 1-tow moderate line speed	35	8.9	1.82	7.20	2.04	1.77
	45	11.8	2.30	7.18	2.03	1.84
	60	14.6	4.30	7.36	2.13	1.77
	80	14.9	4.35	7.44	2.17	1.83
	110	20.3	4.39	7.43	2.17	1.80
MAP 1-tow high line speed	130	13.48	3.12	7.57	2.32	1.80
	160	13.75	3.23	7.62	2.35	1.79–1.86
	180	21.44	5.10	7.68	2.38	1.79
	200	23.81	5.22	7.73	2.39	1.79
Zoltek PANEX 33	—	8.23	1.62	7.37	1.96	1.81
Fortafil 3(C)	—	7.80	1.67	7.43	2.17	1.77

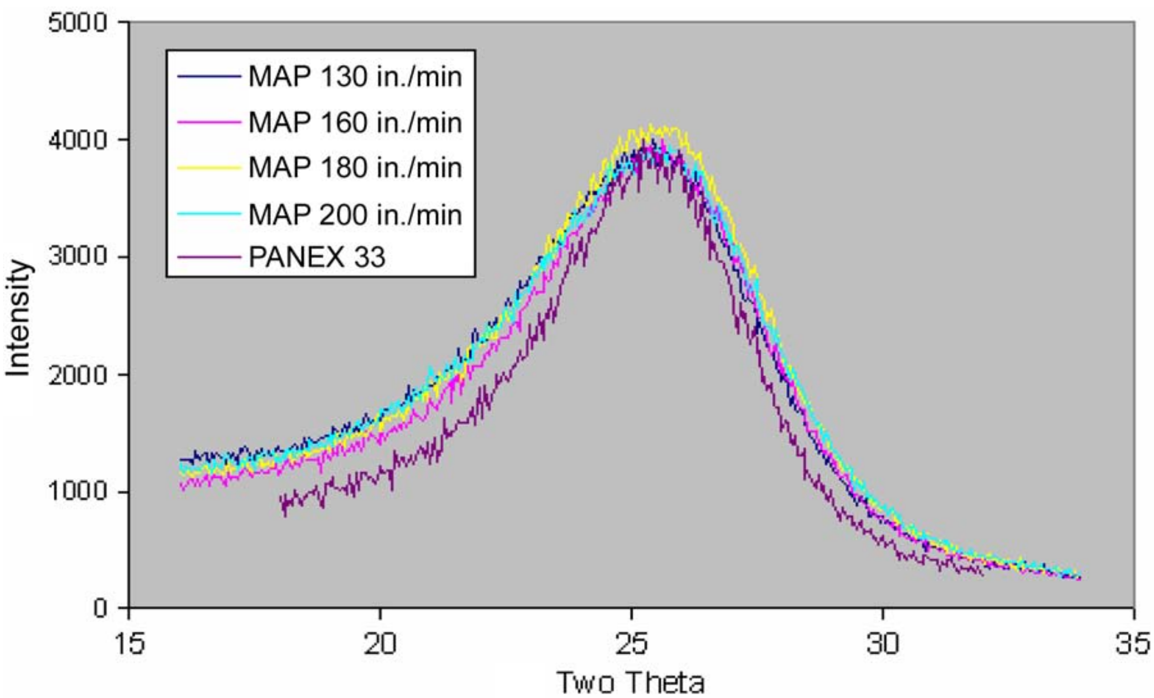

Figure 2. Intensity vs 2θ for the 002 reflection for selected carbon fibers.

the highest single-tow production speeds and compares them to data for the commercial Zoltek Panex 33 fiber. The position of the peak determines the (002) d-spacing, while the breadth of the peak is inversely related to the 'stack height', L_C, of the graphene planes. The plot illustrates that the d-spacings and L_C values are virtually the same, within our ability to measure them, for the four MAP fibers. The values of the d-spacings for these fibers are quite similar to that of the PANEX 33 fiber, but the values of L_C are lower than that of the PANEX 33 fiber. These values are given in Table 2 for these fibers as well as other MAP and commercial fibers. Although there is some scatter in the data, it appears that there is a small, but detectable, tendency for L_C to decrease with increasing production

Table 2. Comparison of MAP and commercial fiber morphologies

Sample description	Misorientation angle[a] (deg)	(002) d-spacing (Å)	Stack height,[b] L_c (Å)	In-plane crystal size,[c] L_a (Å)
MAP 7.5 in./min	19.3	3.465	16.4	39
MAP, 20 in./min	17.4	3.492	16.7	43
MAP 35 in./min	18.5	3.465	17.6	47
MAP, 45 in./min	18.0	3.485	15.2	39
MAP, 60 in./min	18.8	3.453	14.5	35
MAP, 80 in./min	18.9	3.455	14.2	36
MAP, 110 in./min	19.0	3.460	14.2	35
MAP 130 in./min	18.2	3.470	14.7	36
MAP 160 in./min	19.6	3.449	14.4	34
MAP 180 in./min	19.9	3.454	14.4	34
MAP 200 in./min	21.2	3.453	13.9	34
Fortafil 3(C)	19.9	3.503	17.1	45
Zoltek Panex 33	19.1	3.483	17.5	50
Hexcel IM7[d]	15.3	3.495	15.8	44
Thornel P120[e]	3.3	3.378	283	435

[a]Average angle of the graphene planes relative to the fiber axis, as measured by the half-width of the 002 planes in the azimuthal direction.
[b]Average stack height of the graphene planes.
[c]Average crystal size in the direction parallel to the graphene planes.
[d]Aerospace-grade, intermediate-modulus, PAN-based fibers.
[e]Aerospace-grade, high-modulus, pitch-based fibers.

speed of the MAP fibers. This seems reasonable, since increased production speed corresponds to less time at temperature and less time for improvement in the graphene plane stacking. A similar behavior is also observed for the crystal size parallel to the graphene planes, L_a, measured from the breadth of the 100 reflection. The data in Table 3 also shows that the misorientation angle of the graphene planes tends to increase slightly with single-tow production speed of the MAP fibers, but they remain about the same as that of the Panex 33 and Fortafil 3(C) fibers at the highest production speeds.

The tilt angle of the graphene planes with respect to fiber axis is shown in Figure 3,

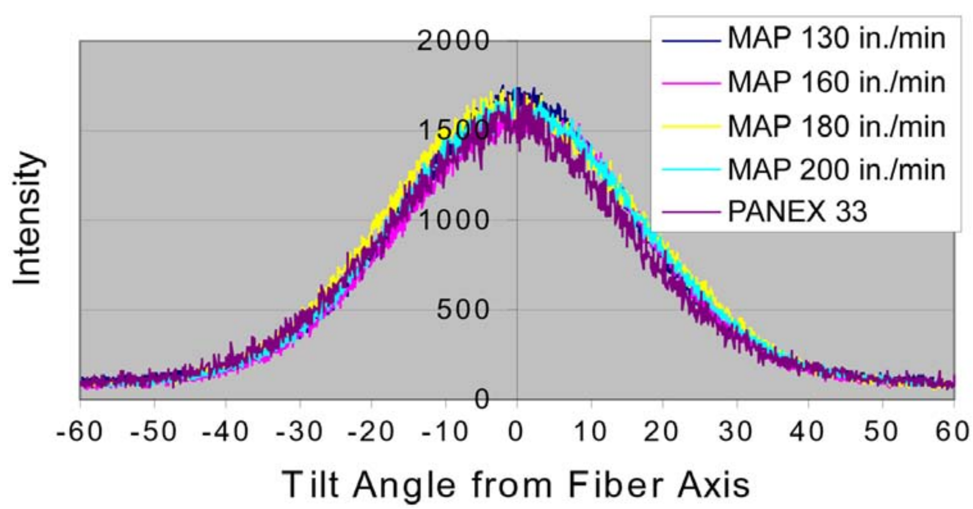

Figure 3. Graphene plane orientation data for selected carbon fibers.

where the distribution of tilt angles is the same for all samples. Slight differences in the average tilt angle can be detected upon careful examination of the data for the individual samples. These differences are reflected in the values recorded for each sample in Table 2. It appears that the primary factor determining the final orientation of the graphene planes is the degree of orientation in the PAN precursor, though there is a very slight tendency for decrease in orientation (greater tilt angles) with increase in processing speed.

Throughput

The economics "figure of merit" is cost per unit throughput. Increasing throughput usually reduces unit cost. Hence the MAP process must be operable at production line speeds and "bandwidth" (number of tows) comparable to or exceeding those of conventional fiber manufacturing lines if it is to realize the desired cost effect. Conventional large-tow carbon fiber manufacturing lines normally run at line speeds in the 90 to 120-in./min range, with about 100 tows per line (a typical line is rated at nominally 1M lb/year when processing 50-K tows).

At the beginning of FY 2003, the researchers had demonstrated a MAP production line speed of 110–120 in./min. In November 2003, the researchers conducted the final experiments on the single-tow line, achieving speeds up to 200 in./min. The researchers speculate, based on their discoveries, that they can substantially exceed 200 in./min on a single-tow line with proper equipment upgrades. However, the decision was made at this point that the project should shift its focus from increasing line speed to increasing the line tow count. Hence the single-tow line was dismantled, and the equipment was reused to construct a three-tow line.

Scale-Up to Multiple Tows

For scaling to multiple-tow processing, a "mitered" MAP applicator design was selected. The "mitered" MAP applicator is similar to the single-tow MAP system except for dimensional scaling and waveguide applicator. The waveguide applicator was redesigned to improve the power distribution profile. An improved waveguide tuner was also built to reduce the anticipated reflected power from the new applicator. The new three-tow feedthrough system is conceptually identical to that used in the single-tow MAP system, but details were revised to improve sealing.

In May, the three-tow mitered MAP system was operated for the first time. Initial operations utilized finished tows to test and adjust mechanical parameters such as tow transport, tensioning, and sealing, at 13-in./min line speed. Plasma conditions were also monitored; however, significant differences in the properties of oxidized and finished fibers limited the value of observations about the plasma behavior. Observed parameters were consistent with expectations during operation of the three-tow mitered MAP system using standard fully oxidized tows, also at 13 in./min line speed. Background pressure was satisfactory, and the plasma power distribution was improved. Measured tow linear resistance was satisfactory. In August the researchers conducted a three-tow run at 12 in./min. to produce a quantity of carbon fiber sufficient for mechanical testing. Excellent mechanical properties resulted, as shown in Figure 1. A stable operating mode that requires very little feed gas was unexpectedly discovered during this experiment. During attempts to run the three-tow line at a higher speed, the reflected power rapidly rose too high, limiting the maximum line speed to 13 in./min. Improved component matching is needed to push the system to higher line speeds, and the necessary design modifications are under way.

Parametric Studies

During the final single-tow MAP high-speed run, a series of microwave electric field measurements were made along the waveguide applicator to understand how the microwave power, and therefore the plasma power, is distributed along the tow. The results indicate that the plasma power drops off rapidly from the microwave feed point (tow output) and then levels out midway along the length of the waveguide applicator to the other end where the tow is introduced. Superimposed on this variation is a standing wave pattern. Therefore, a point on the moving tow is subjected to an alternating series of high- and low-power processing steps that rapidly increase until that point on the tow leaves the chamber at the microwave feed point.

Instrumentation and Control

Further evaluations were undertaken to calibrate the high-temperature resonance cavity for in-line, real-time dielectric measurement of carbon-fiber tows, with the goal of elevating the cavity's maximum service temperature from ~500°C to ~1,000°C. Because the carbon fibers become very electrically conductive at high temperatures, the resonance cavity must necessarily be very sensitive at high temperatures. The high-temperature resonance cavity was electroplated. The conventional oven and required temperature controls were evaluated. Control software was acquired and customized to closely mimic the real production temperature profile during any stage of processing. The data acquisition software was installed and tested. The medium-temperature system for dielectric measurements of the carbon-fiber tows, from oxidized state to 500°C, is ready for functional testing.

New tow winding and tensioning equipment was installed on the multiple-tow processing line as follows:

- Designed for 200 in./min and readily upgradeable to 400-in./min line speed.
- Designed for 50-K nominal tow size, but can handle tow sizes ranging from 3 K to 64 K with minor modifications to the processing line. Up to three different tow sizes can be processed simultaneously.
- Triple tension-controlled creel for independently controlling tension in three tows.
- Triple take-up winder with feed roller and independent speed control. It provides very well-controlled constant speed, independently controllable for each tow, during processing. This permits simultaneous evaluation of different processing conditions for each tow, e.g., different tension levels, line speeds, and tow size during a single experiment.

Patents

Patent No. 6,514,449 B1, entitled "Microwave and Plasma-Assisted Modification of Composite Fiber Surface Topography," issued February 4, 2003.

Spin-Offs

A number of "spin-off" developments have resulted from this project. Work in the following areas was conducted at a low level of effort as part of this project during this reporting period.

- **Carbon-fiber recycling—**Performed X-ray photoelectron spectroscopy (XPS) and scanning electron microscope (SEM) evaluation and characterization of fibers that were recovered in ANL recovery process.
- **Surface treatment—**Evaluated and compared surface chemistry and surface roughness of ORNL's MAP fibers and commercial fibers.
- **Storage—**Discovered a way to repeatably produce fibers with a layered morphology that may be useful in gas storage applications.

Education

The materials characterization, notably utilizing SEM and spectroscopy methods such as XPS and secondary ion mass spectrometry (SIMS), has been conducted in partnership with the University of Tennessee's Materials Science Department. Three UT graduate students, one a Ph. D. candidate and two M.S. candidates, were provided characterization support to the project. One M.S. candidate was awarded his degree during this reporting period, based in part upon his contributions to this project.

Conclusions

The development of MAP carbonization and graphitization technology has progressed according to plan. All FY 2003 milestones were satisfied on schedule. Construction and initial operation of a three-tow research line has been successfully completed, with the fibers initially produced therein exhibiting excellent mechanical properties. MAP processing is generally considered to be an evolving "success story" with great potential for making an impact on carbon-fiber technology and cost.

Summary

These are highlights of the progress during FY 2003.

- The continuous single-tow MAP carbon-fiber pilot facility was successfully operated, and 50-K PAN fiber tows were carbonized/graphitized at processing line speeds up to 200 in./min.
- The continuous three-tow MAP carbon-fiber pilot facility was constructed and successfully operated. Three 50-K PAN fiber tows were simultaneously carbonized/graphitized at processing line speeds up to 12 in./min.
- Physical, mechanical, and morphological properties were evaluated for fiber samples manufactured at single-tow line speeds up to 200 in./min. Physical and mechanical properties were measured for fiber samples manufactured at three-tow line speeds up to 12 in./min. In most cases, properties exceeded program requirements.
- We calibrated the improved resonance cavity for a dielectric measurement system that can operate at >1,000°C with kilohertz sampling rates.
- We installed a new three-tow winding and tensioning system designed for line speeds up to 200 in./min, readily upgradeable to 400 in./min, with independent control for each tow.
- One patent was formally issued.